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STUDY OF THE HIGH-TEMPERATURE OXIDATION OF THE SIKHOTE-ALIN' IRON METEORITE

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ABSTRACT. The study made of the high-temperature oxidation of the Sikhote-Alin' iron meteorite material and of an iron-nickel alloy in air at atmospheric pressure, and in an environment with a low partial oxygen pressure, is described.

The authors studied the high-temperature oxidation of material from the Sikhote-Alin' iron meteorite in air at atmospheric pressure and in an environment with a low partial oxygen pressure. Used for the study was material from the Sikhote-Alin' iron meteorite shower (an individual sample was obtained from the Committee on Meteorites of the Academy of Sciences of the USSR through the Ural Commission on Meteorites), as well as an iron-nickel alloy (weight content, in percentage: Ni - 5.96; C - 0.020; Si - 0.014; Mn - none; Cr - none; S - 0.005; Fe - remainder). /60*

Test specimens for long-term heating were made in the form of rectangular wafers, the average dimensions of which were 10 x 10 x 2 mm. The specimens were sanded down and degreased prior to oxidation. Oxidation in air took place in a vertical tube furnace. Specimens were placed in an alundum crucible. Some of the experiments, in which the formation of the crust of fusion was simulated, were carried out by heating in an open induction furnace. Specimens used were in the form of small cubes with a 10 mm edge. Quartz was used as the backing for the specimens. Heating lasted for from 36 to 90 seconds. The maximum temperature achieved at the surface of the specimen was approximately 1600°C. The diffusion zone at the edge of the cube not in contact with the backing was studied.

Specimens were annealed in airtight steel containers filled with ferric oxide (category ChDA) to ozidize them in an environment with a low partial oxygen pressure. The long-term oxidation experiments were carried out in the range

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of temperatures between 700° and 1200°C, with the average duration 10 hours. The reaction diffusion zone was studied by making metallographic and radiographic investigations (using the Debye method for the photography in RKD and KROS-1 chambers).

General Characteristic of the Diffusion Zone

Annealing of the meteorite material, and of the iron-nickel alloy, in the oxidizing environment was done under all of the conditions studied during the work in order to form a diffusion zone in which it would be possible to distinguish two parts: an external, henceforth called the scale part, and an internal, in the form of an oxidized metallic layer that we shall call the subscale part. The scale was weakly bonded to the subscale part in the case of specimens oxided in air, but there was a strong bond between the scale and the subscale when oxidation took place in ferric oxide (Fe_2O_3) powder.

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Figure 1*. Diffusion zone in the iron-nickel (6% Ni) alloy. Oxidation in air. Annealed for 10 hours at 1000° x 200. I - scale; II - subscale; III - original alloy.

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* Figure not reproducible.

Figure 1 is included as an example of the microphotography of the diffusion zone in the iron-nickel alloy. The diffusion zone in the meteorite material has a similar structure, but the preparation of the thin sections was extremely difficult because of the poor bond between the scale and subscale.

Scale Structure and Phase Composition

Phases characteristic of pure iron scale, wüstite (FeO), magnetite (Fe_3O_4) and hematite (Fe_2O_3), can be observed radiographically in the powdered scale, as well as by layer radiography. This does not preclude the possibility that these phases are, to a certain extent, alloyed with nickel. However, the radiography procedure we used did not provide the opportunity to establish the variations in the parameters of the crystal lattice needed to provide a basis for determining the degree of alloying. Observation of the alloying ability of the spinel phase is difficult because of the proximity of the magnetite and nickel ferrite parameters. Radiographic observation of the alloying ability of wüstite too is difficult because of the considerable change that takes place in the parameters of the wüstite lattice in the region of its homogeneity [1]. The oxide phases of nickel in the diffusion zone were not observed, radiographically, or metallographically. Figure 2 is a microphotograph of scale forming on the iron-nickel alloy after 10 hours of annealing in air at 1100°C. Its complex structure can be seen quite clearly. The outer layer is hematite, the middle magnetite, and the inner a two-phase layer, apparently a mixture of magnetite and nickel ferrite. The two-phase nature of the layer is seen quite well on the microsection at the same time that one phase with a spinel type lattice is exposed radiographically.

Subscale Structure /62

The subscale is the most interesting, and at the same time the most complex in structure. It also is the most sensitive to change in oxidation conditions and composition of material.

Figure 2*. Scale on the iron-nickel alloy (6% Ni). Annealed in air for 10 hours at 1100°. x 200. I - hematite; II - magnetite; III - magnetite and nickel ferrite.

Figure 3*. Meteorite (a) and iron-nickel alloy (b) subscale. Annealed in air for 10 hours at 1100°. x 200.

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* Figures 2 and 3 not reproducible.

Figure 3 shows microphotography of a transverse section of meteorite subscale oxidized in air at 1100°C (Figure 3a), and of the iron-nickel alloy (Figure 3b). The subscale in both cases has the same structure, generally speaking, as will be seen from the photographs, and is a mixture of the oxide and metal phases. The magnitude of the dispersion of the metal phase increases as the scale approaches the edge.

Layer radiography of the subscale revealed that it is a mixture of wüstite and the metal phase with a face-centered lattice, the parameter of which is close to the nickel parameter. Nevertheless, there is a definite difference between the structure of the subscale of these two materials. The oxidation front of the alloy is quite uniform when it oxidizes, whereas the grain boundaries are involved first when the meteorite is oxidized.

The structure of the subscale is quite different when oxidation takes place in ferric oxide powder, that is, under low partial oxygen pressures. In this case, the oxide phase (the wüstite) is dispersed in the form of granules, or seams, in the metal phase with a face-centered cubic lattice, that is, nickel, or a solid solution of iron in nickel, is not seen radiographically during oxidation in powder.

The metal phase in the region contiguous to the scale has a tendency to form a complex layer during oxidation of the meteorite. This layer is clearly defined during oxidation of the alloy. Dispersions of the oxide phase are separated from the layer of scale by a layer of metal when the concentration of nickel is increased.

Crust of Fusion Structure

The study of the structure of the diffusion zone upon short-duration heating of the meteorite material in air to a temperature above the melting point of oxides of iron is interesting. Experiments such as these definitely simulate the formation of the crust of fusion on the meteorite.

Figure 4*. Diffusion zone upon heating for 90 seconds. a - scale with magnetite dendrites; b - subscale. x 200.

Figure 5*. Diffusion zone upon heating for 35 seconds. x 300.

* Figures 4 and 5 not reproducible.

Figures 4 and 5 are the corresponding microphotographs. We see from them that in the case of short-duration heating (35 to 90 seconds) to high temperature /64 (1500°C) the diffusion zone is divided into two parts that differ sharply in structure; the scale, and the subscale. The scale has the same composition when heated for a short period of time to high temperatures as it does after long-term oxidation in air. However, the scale structure contains complications occasioned by the melting of the oxide components.

The presence of fusion of the scale can be judged from the dendrites and the round inclusions of magnetite after heating for 35 seconds (Figure 5). A film of metallic nickel forms along the boundary between the scale and the subscale. This is exposed radiographically. The subscale beneath the nickel film is a mixture of wüstite and nickel.

Metallic nickel can be observed in the subscale after oxidation for 35 seconds. The scale basically is wüstite with circular dispersions of magnetite.

Discussion of the Results

The experiments revealed that high-temperature oxidation of the Sikhote-Alin' meteorite under laboratory conditions takes place in the same general way that oxidation of the iron-nickel alloy does. The information on the structure of the diffusion zone upon oxidation of the meteorite material and of the alloy with 6 percent Ni in air at atmospheric pressure obtained during the work agrees well with the data obtained by J. Bénard and J. Moreau [1] with respect to the oxidation of pure iron-nickel alloys. The literature contains no information on the oxidation of iron-nickel alloys at low partial pressures, when only the lowest oxide of iron, wüstite, forms. Nevertheless, our experiments suggest that the structure of the diffusion zone in this case differs from the structure of the diffusion zone when oxidation takes place in air.

Nickel forms in the subscale upon oxidation in air at atmospheric pressure, and the metallic component consists of nickelous iron when oxidation takes place in ferric oxide powder. According to J. Bénard [2], the formation of nickel results from a secondary reaction between the wüstite, the impoverished oxygen, and the nickel oxide, and is possible because of the existence of a state with a higher valency of the alloy's main component, iron. This same type of reaction should take place upon oxidation in the Fe_2O_3 powder, but was not observed. In this case, the metallic phase in the subscale consisted of nickelous iron. This fact indicates that the mechanism involved in the oxidation of iron-nickel alloys, and of the meteorite material, is different from that indicated by the authors of reference [1]. It is not yet possible to provide an exhaustive explanation of the mechanism involved in the oxidation of the alloys discussed, but its general outlines are as follows.

The alloying element (nickel) has less affinity for oxygen than does the base metal, and the nickel oxide correspondingly has a higher disassociation pressure than does wüstite. Consequently, when the alloy contains a concentration gradient the wüstite can occur in those regions where it still is impossible for nickel oxide to form. There is a simultaneous enrichment of the alloy with the alloying element.

This explanation requires a definite solubility of the oxygen in the alloy, and a low diffusion coefficient (as compared with iron), as well as a broad concentration region on the Fe-Ni-O diagram, where the wüstite can be found in equilibrium with the solid solution of iron and nickel.

Oxidation along the grain boundaries in the meteorite is more sharply defined than is the case in the alloy. This is true for all conditions, and apparently is the result of the influence exerted by the impurities.

V. D. Kolomenskiy and I. A. Yudin studied the structure of the crust of fusion of the Sinkhote-Alin' meteorite [3]. Metallic nickel was not found in the crust, only nickelous iron. But we, in our experiments, observed inclusions of metallic nickel, even during short intervals of annealing at a temperature higher than the melting point of the iron's oxide phases. These discrepencies can be explained, possibly, by the fact that an air cushion with a reduced partial oxygen pressure was formed during the meteorite's flight.

In conclusion, we consider it to be our duty to express our appreciation to I. A. Yudin for his constant attention to the work, and to F. A. Sidorenko for his assistance in conducting the experiments.

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